OXFORD CRYOSYSTEMS

PheniX

Operation & Instruction Guide

OXFORD CRYOSYSTEMS PHENIX

Operation & Instruction Guide v2.6

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1 Introduction

The PheniX cryostat is designed for the cooling of flat plate powder samples down to temperatures of around 12 K. The PheniX is designed to fit to vertical powder diffractometers and be used in both the theta - theta and theta - 2 theta modes. This is made possible by a uniquely designed rotating seal between the integrated Gifford-McMahon Cryocooler and the sample stage. This allows the sample stage to move in the full 2-theta range of the detector without the high pressure hoses and cables becoming tangled up or interfering with other services in the enclosure.

The compact design means the PheniX can be easily fitted to the goniometer from the front with a specific adapter plate designed for each type of goniometer.

1.1. How the PheniX works

The gas flow circuit of the PheniX is shown in Figure 1 below.

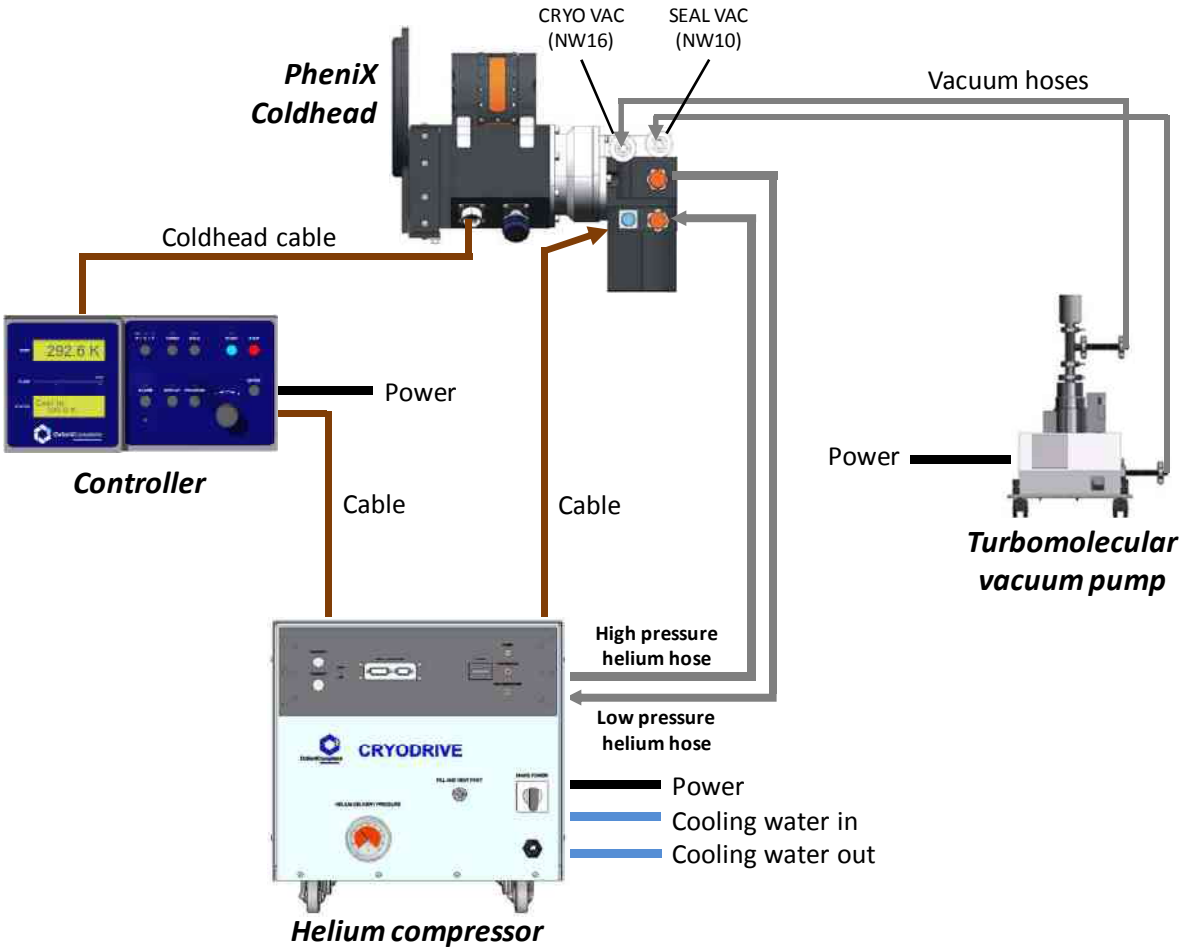


Figure 1 – PheniX layout

The closed cycle cooler is mounted within the body of the PheniX and operates using compressed helium gas provided by the Cryodrive compressor, which is water-cooled. It is important to note that there is no helium gas consumption in this system and the helium gas circuit in the Cryodrive/Coldhead combination is sealed. The sample stage is cooled by the conduction of heat between the sample stage and the cold stages of the Coldhead.

A Vacuum System is used to continuously pump the vacuum space around the PheniX internals and also the Rotating Seal to minimise unwanted heat leaks into the system. It is important that a good vacuum ($<10^{-4}$ mbar) is maintained with the Coldhead otherwise achieving extremely low temperatures at the sample becomes impossible.

The PheniX can cool the sample stage to temperatures as low as 12 K and as high as 310 K with a stability of better than 0.1 K.

1.2. Items required for assembling your PheniX

The component parts of the system are:

- The PheniX Coldhead
- 730 Series Controller
- Grey Cryodrive cable
- Black Coldhead cable
- Cryodrive compressor
- 2x Stainless steel flexible transfer lines
- Pfeiffer Turbomolecular Vacuum Pump
- CD with Operating Manuals and Cryopad software
- PheniX Mounting Plate for Goniometer

2 Setting Up

2.1. Matching the PheniX Coldhead and 730 Series Controller

The PheniX Coldhead and 730 Series Controller are supplied as a matched pair. The individual characteristics of the Coldhead sensors are programmed into the Controller. Please contact your supplier if you believe you may have unmatched units.

2.2. Operating voltage

It is essential that the PheniX is configured to operate on the local mains electrical supply.

Item	Operating voltage requirements
730 Series Controller	The voltage selector switch on the rear panel must be set to the correct position: 200-240 Volts AC, 50 Hz, 3 Amps 100-120 Volts AC, 50-60 Hz, 6 Amps. Make sure an 'Anti-Surge' (T) type fuse of the correct rating is fitted.
Cryodrive	User configurable Supply voltage: 200, 220 or 240 V at 50 Hz or 200, 208 or 220 V at 60 Hz Supply voltage tolerance: +10 % Maximum supply fuse rating: 30 A Recommended CryoController fuse type: Slow blow Maximum CryoController fuse rating: 5 A Over voltage category (IEC664): 2
Pfeiffer Turbomolecular Vacuum Pump	90-132 V 50/60 Hz and 185-265 V 50/60 Hz. This pump unit will automatically run on the correct voltage.

Oxford Cryosystems ships all Cryodrives set to 240 V and 50 Hz as this is how they are commissioned in the UK. It will be necessary to refer to Section 2.3 to set the Primary Tap Connections for the local voltage and frequency.

2.3. Electrical supply connection for Cryodrive

We recommend that you use a suitably fused isolator at your electrical supply outlet. Locate the isolator switch close to the electrical outlet. It is also recommended that back-up fuses are installed at the electrical supply outlet. You must configure the Cryodrive to suit your electrical supply. The Cryodrive is despatched configured for use with 240 V and 50 Hz electrical supply.

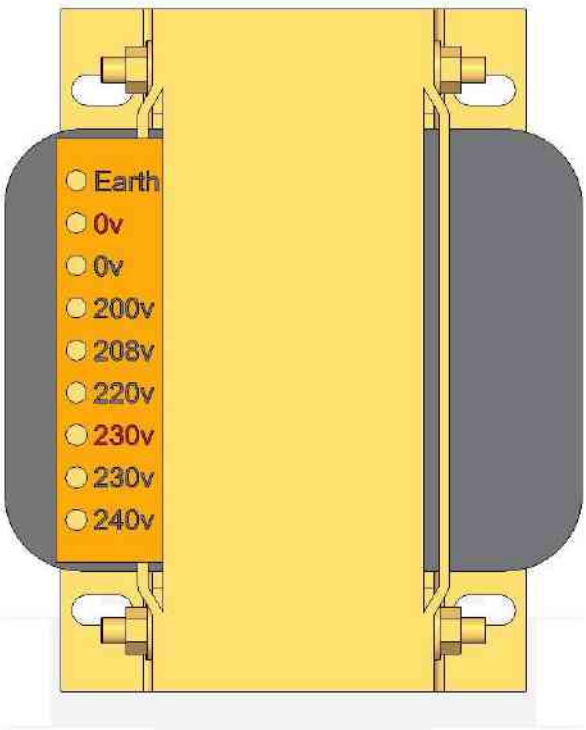


Figure 2 – Transformer showing different electrical supply connections

Use the procedure below to change this configuration.

- Look at Table 1: find your electrical supply in the left-hand column, look along this row to find the connections you must make for the wire 30 (W30) and wire 31 (W31).

- Remove the lid of the Cryodrive and locate the transformer.

- With reference to Figure 2, change the position of wire 30 (W30) and wire 31 (W31) so that they are in the correct position for your electrical supply. Ensure the connections are fully tightened. **Do not move the wires attached to the red 0 V and 230 V connectors, as these provide power to the secondary stepper motor transformer and are not part of the procedure.**

- Locate the protection switch on the current-limit potentiometer which is situated in the CryoController, refer to Figure 3. The switch can be accessed by removing the lid of the CryoController unit.

- Look at Table 2: find your Cryodrive type and electrical supply frequency in the left-hand column, look along this row to find the recommended limit for the protection switch current.

- Use a small screwdriver to adjust the current-limit potentiometer to the recommended value.

- Replace the lid of CryoController and the Cryodrive.

- Make sure that the Cryodrive ON/OFF switch is in the OFF position and connect the Cryodrive to your electrical supply.

Electrical Supply	Primary Tap Connection					
	N	200 V	208 V	220 V	230 V	240 V
50 Hz, 200 V	W32	W31	W30	-	-	-
50 Hz, 220 V	W32	-	W30	W31	-	-
50 Hz, 240 V	W32	-	W30	-	-	W31
60 Hz, 200 V	W32	W31	-	-	W30	-
60 Hz, 208 V	W32	-	W31	-	W30	-
60 Hz, 220 V	W32	-	-	W31	W30	-
60 Hz, 240 V	W32				W30	W31

Table 1 – Wire connections to primary tap connections

Cryodrive model	Electrical supply voltage			
	200 V	208 V	220 V	240 V
Cryodrive 1.5, 50 Hz supply	11 A	-	10 A	10 A
Cryodrive 1.5, 60 Hz supply	11 A	11 A	11 A	-
Cryodrive 3.0, 50 Hz supply	16 A	-	16 A	16 A
Cryodrive 3.0, 60 Hz supply	18 A	17 A	16 A	-

Table 2 – Recommended protection switch current limit setting

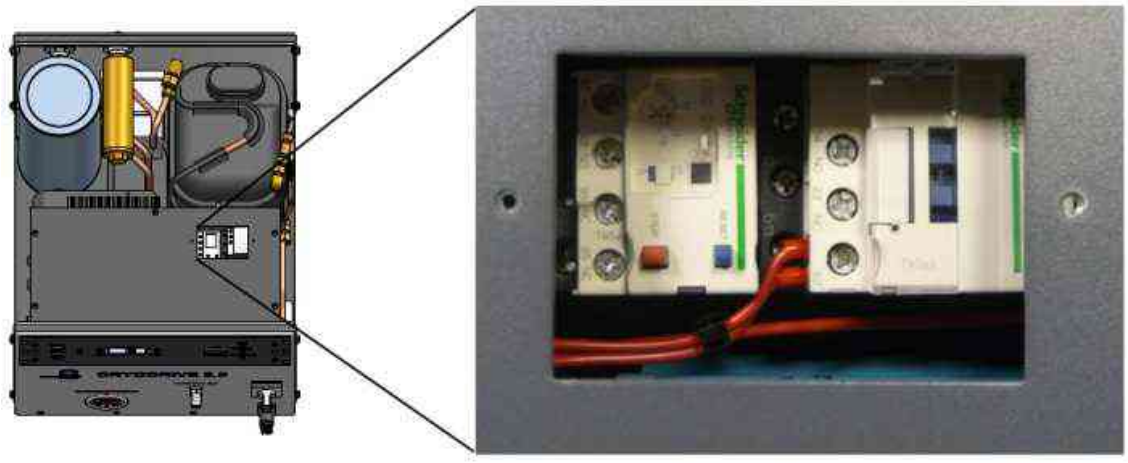


Figure 3 – Overload relay showing stop and reset buttons

2.4. Layout of Cryodrive

Figure 4 shows the layout of the Cryodrive.

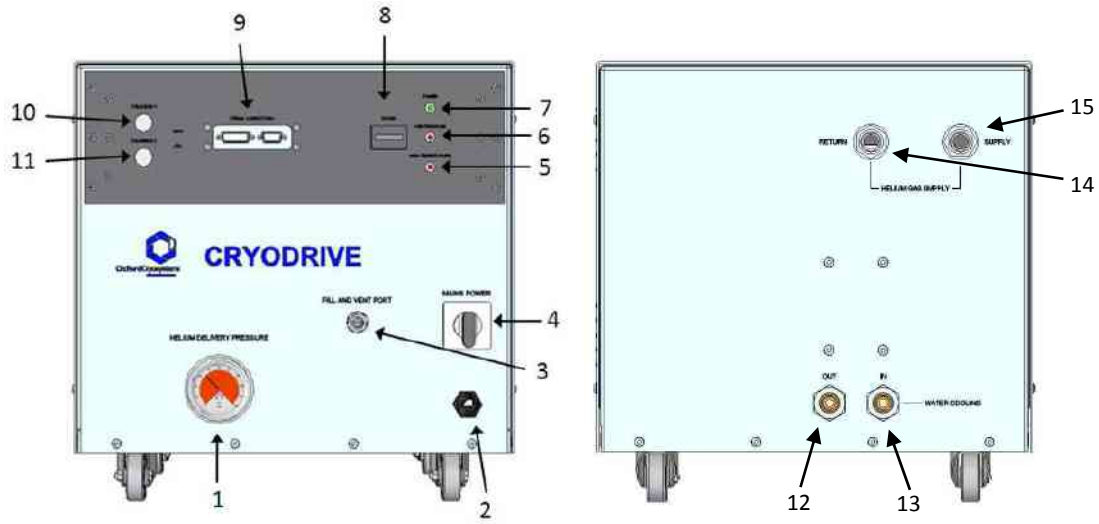


Figure 4 – Front and back of Cryodrive

1. Pressure gauge
2. Mains supply in
3. Charge and vent port
4. ON/OFF (reset) switch
5. High temperature LED
6. Low pressure LED
7. Cryodrive ON LED
8. Elapsed hour counter
9. Serial cable connections
10. Coldhead 1 connection
11. Coldhead 2 connection
12. Cooling water outlet
13. Cooling water inlet
14. Helium return
15. Helium supply

2.5. Helium gas hose connection

WARNING

If you are unsure on how to connect the helium gas hoses, please contact Oxford Cryosystems. Failure to connect the hoses properly might result in complete loss of helium pressure within the Cryodrive.

The main layout is illustrated schematically on the diagram in Figure 1 and connections are generally labelled equivalently at each end. Start with the two high-pressure hoses between the PheniX Cooler and the Cryodrive compressor module. The ends are colour coded to avoid confusion: the helium return line has a GREEN band; helium supply line has a RED band, see Figure 5.

Figure 5 – Helium gas hoses

The Cryodrive has one helium supply outlet and one helium return inlet. The recommended procedures for connecting and disconnecting the couplings are described below (for more information read the Cryodrive Operation & Instruction Guide).

1. **To prevent damage to the couplings and leakage of the helium, you must use two spanners as shown in Figure 6**.
2. Complete the fitting as quickly as possible to prevent leakage of the helium from the couplings.
3. Note that helium hoses are pressurized with helium. When you fit these components, you must follow the safety advice and instructions given in the instruction manual supplied with them. Do not over bend or twist the helium hoses; do not allow damage to occur to the braid on the outside of the hoses.
4. Connect the hose marked with the red band to the helium supply connector on the Cryodrive. Connect the other end of the hose to the helium supply connector (marked with a red band) on the PheniX Cooler.
5. Connect the hose marked with the green band to the helium return connector on the Cryodrive. Connect the other end of the hose to the helium return connector (marked with a green band) on the PheniX Cooler.
6. Check that the connecting surfaces of the couplings are clean.
7. Check that the sealing 'O' ring is in place.
8. Connect the coupling halves by hand until you feel resistance.
9. Refer to the image below and rotate spanner (green arrow) whilst holding the other spanner still (red arrow) to fully tighten the coupling.
10. When tightening the hoses both at the Cryodrive and the PheniX coldhead, ensure the fittings are tightened until a good resistance is felt. In the case of the Cryodrive fittings, the helium connections should be tightened so that the two faces of the male and female fittings meet. On the coldhead itself, the faces of the fittings will not meet but in order to ensure the coupling is open make sure a good resistance is felt. A helpful indication is that approximately 12 mm of thread on the male fitting can be seen when the coupling is properly tightened.

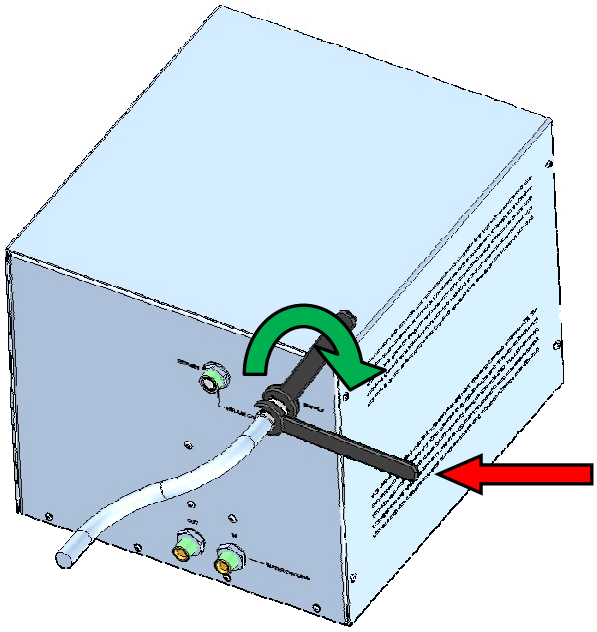


Figure 6 – Connection of a helium hose to the Cryodrive using two spanners

NOTE

When the connection is fully made, rotate one spanner in the opposite direction for one quarter turn to ensure that the sealing 'O' ring is not over compressed. If you over compress the sealing 'O' ring the service life of the fittings will be reduced.

2.5.1. Helium gas pressure check

The location of the pressure gauge is illustrated in Figure 7. Whilst the Cryodrive is running, the pressure indicated is significantly higher than the quiescent value of approximately 16.5 bar. This is best shown by a red label with a black line marking 16.5 bar. It is important to read the gauge at eye level to ensure that pressure is at 16.5 bar. Under normal operating conditions it should be about 22 bar and will noticeably oscillate by around 0.5 bar. It is good practice to monitor the Cryodrive charge pressure, especially if the system has been dismantled and reconnected recently, as gas can be lost during the attachment and disconnection of the high-pressure hoses. As mentioned before, if the pressure is observed to have fallen, contact Oxford Cryosystems to arrange a re-charge.

IMPORTANT – If the Cryodrive is used for the first time

It is important to check the charge pressure of the Cryodrive compressor. In the quiescent state, the pressure should read approximately 16.5 bar (240 psi). If this pressure reads less than 15.5 bar (220 psi), contact Oxford Cryosystems. This check should be repeated, if possible, each time the unit is to be turned on. Under normal operating conditions it should be about 22 bar and will noticeably oscillate by around 0.5 bar. It is good practice to monitor the Cryodrive charge pressure, especially if the system has been dismantled and reconnected recently, as gas can be lost during the attachment and disconnection of the high-pressure hoses.

Figure 7 – Front of Cryodrive showing the pressure gauge

2.5.2. Cryodrive and Controller Cable Connections

Connect the PheniX Coldhead cable connector from the Cryodrive to the bayonet fitting on the Cryocooler Coldhead of the PheniX.

Connect the serial cable from the Cryodrive to the 'Cryodrive' socket on the back of the PheniX Controller.

Connect the PheniX Coldhead Cable from the 'Coldhead' socket on the back of the Controller to the multi-pin bayonet socket on the side of the PheniX cryostat body located next to the relief valve.

Connect the separately supplied Cryopad serial cable from the 'Serial' socket on the back of the PheniX Controller to an appropriate COM port on a PC.

The PheniX Controller requires connecting to power using the IC power cable supplied.

2.5.3. Cooling water connection

1. Use hose clips to secure suitable water hoses (1/2-inch nominal internal diameter) to the water connection nozzles.

2. Connect the supply and return hoses to the cooling water inlet and outlet connectors as marked on the rear of the Cryodrive.

3. Connect the water supply hose to a cooling water supply with an adequate flow rate and temperature (see section 2.5.4).

4. Connect the water return hose to a suitable drain.

5. Turn on the cooling water supply and check that there are no water leaks.

2.5.4. Water cooling requirement

- Water cooling/chiller requirement: 3.0 kW
- Minimum flow rate: 1.5 L/min
- Maximum flow rate: 7.0 L/min
- Maximum water supply pressure: 101.5 psig.
- Minimum water supply temperature (at start –up): +4ºC.
- Maximum water discharge temperature: +33ºC.
- Water quality pH range: 6.0 to 8.0.
- Maximum calcium carbonate concentration: 75 ppm.

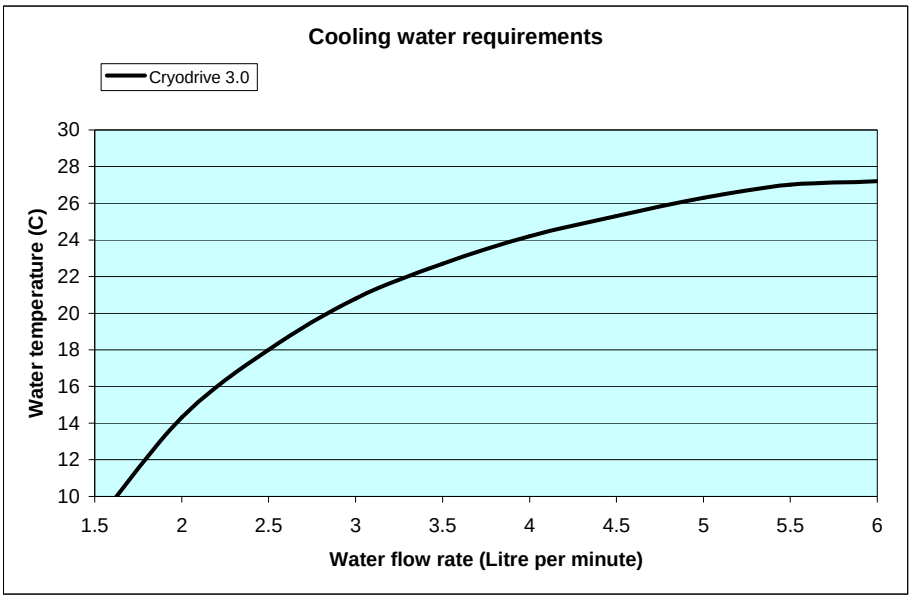


Figure 8 – Cooling water requirements

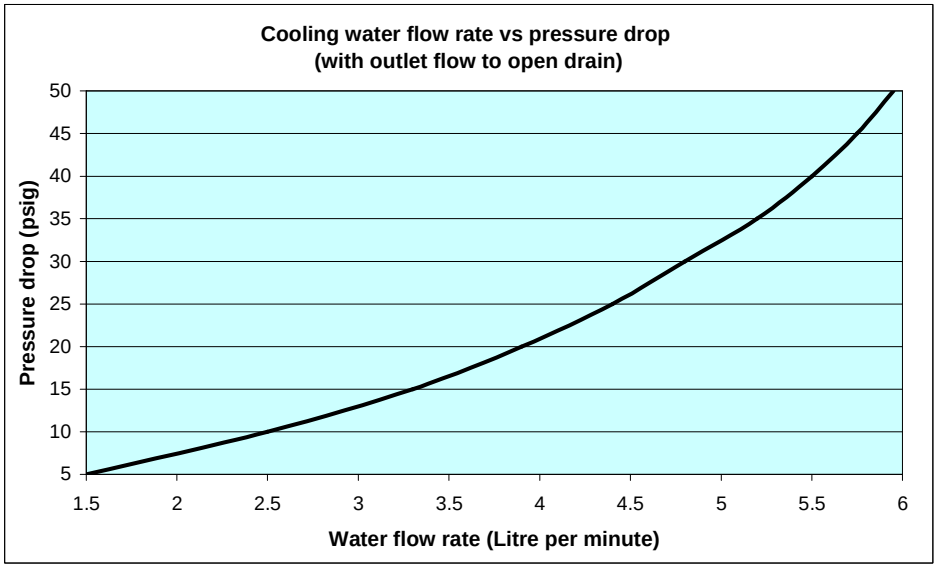


Figure 9 – Cooling water flow rate vs pressure drop

2.6. Vacuum System

Oxford Cryosystems currently provide the Pfeiffer Turbomolecular Vacuum Pumping Station shown in Figure 10. If an alternative turbo pumping station is to be used, please refer to the relevant manufacturer's instruction manuals.

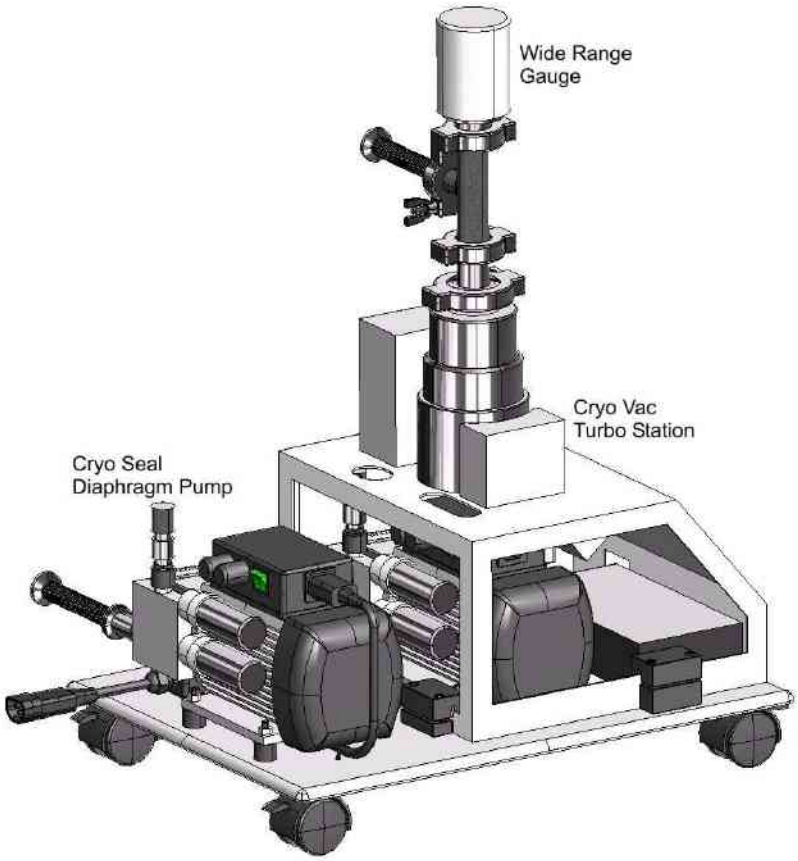


Figure 10 – PheniX Vacuum Pump Station

NOTE

A variety of manuals are supplied with the Pfeiffer Vacuum System for all its components. The information below outlines the simple steps needed to use this system with the PheniX. Further information is available in the manuals, if necessary.

WARNING

Before installation of the Vacuum System we urge you to read all the Operating Instructions supplied with the Pfeiffer Vacuum System for all its components. You will find instructions regarding the safety, storage and installation which must be followed to prevent damage to the equipment or personnel.

2.6.1. Connecting up the Vacuum System

NOTE

When assembling the vacuum components ensure that all vacuum sealing surfaces are free from dust to guarantee a good vacuum seal.

1. Remove any packing material from around the Vacuum System.

2. Remove the dust cap from the top of the Turbo pump.

3. Fit the Meshed Centring Ring, 'T' piece and NW40-25 reducer on the top of the Turbo pump using one of the NW25 clamps and centring rings supplied.

4. Fit the Wide Range Gauge to the top of the 'T' piece using one of the NW25 clamps and centring rings provided.

5. Fit the NW16 flexible hose to the remaining NW16 fitting on the 'T' piece using a clamp and centring ring.

6. Using an NW10/16 clamp and centring ring, fit the NW10 flexible vacuum hose to the port on the Cryo Seal Diaphragm Pump.

7. Using an NW10/16 clamp and NW16 centring ring, fit the NW16 flexible vacuum hose from the top of the Turbo T piece to the Cryo Vac port on the PheniX.

8. Using an NW10/16 clamp and NW10-16 centring ring, fit the NW10 flexible vacuum hose from the Cryo Seal Diaphragm Pump to the Cryo Seal port on the PheniX.

The Vacuum System is designed as a user-friendly system, which is straightforward to operate. The unit comprises a small Diaphragm Pump and a Turbo Station with an additional integrated Diaphragm Pump along with a Wide Range Gauge and a Display and Operating Unit:

1. Cryo Vac Turbo Station and integrated Diaphragm Pump - this turbo system pumps the main body of the PheniX.

2. Seal Vac Diaphragm Pump - this pump evacuates the rotating seal of the PheniX to ensure that when the seal moves, the main body vacuum is then unaffected.

Both of these pumps are independent of one another. Although the mains power is fed through a common junction box, both the Cryo Vac Turbo Station and Seal Vac Diaphragm Pump have separate power switches as indicated in the pictures below.

Power Switch for Seal Vac Diaphragm Pump

Power Switch for Cryo Vac Turbo Station and integrated Diaphragm Pump

As outlined above, unpack the Vacuum System and ensure that all packing material has been removed from around the unit including from around the integrated Diaphragm Backing Pump inside the Cryo Vac Turbo Station. Fit the Wide Range Gauge head and 'T' assembly to the top of the Turbo Station and using the supplied vacuum hoses and fittings, connect the vacuum lines from the Cryo Vac Turbo Station and the Seal Vac Diaphragm Pump to the PheniX. This is normally performed by the Installation Engineer.

2.6.2. Switching On the Seal Vac Diaphragm Pump

Once the unit is connected up, switch the pump on using the green power switch. The pumping of air can cause moisture/vapour to condense in the pump and can have an effect on the attainable final pressure. To expel these condensates, gas ballast air can be admitted to help obtain these final pressures. To ensure a good vacuum, open both Gas Ballast Valves for an hour during initial pumping to ensure any residual water vapour and air in the lines and PheniX are removed. Figure 11 below illustrates how sleeve '43' should be moved to position 'Y' to open the gas ballast valve. Be sure not to leave the valve open permanently. **Note: Always remember to close the ballast valve after use.**

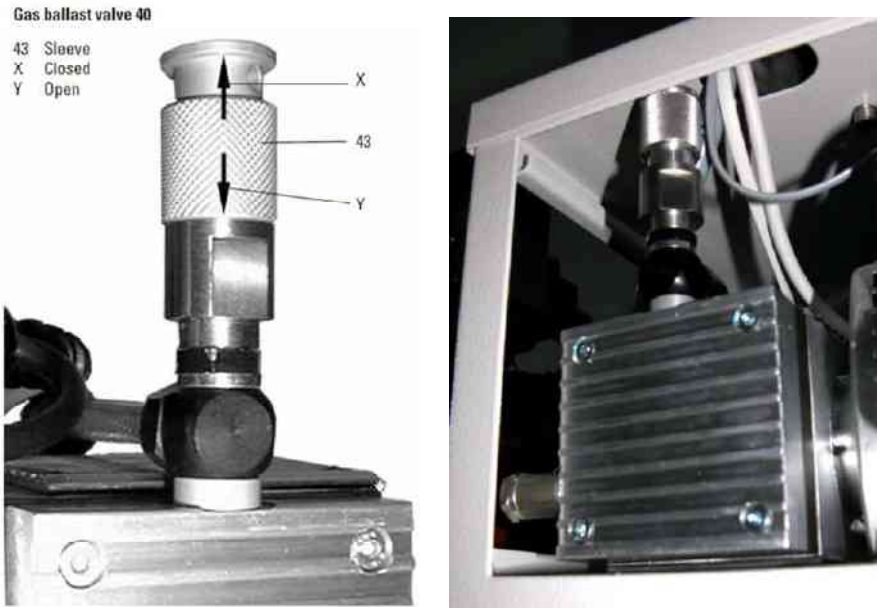


Figure 11 – Ballast valve in the closed position.

2.6.3. Switching On the Cryo Vac Turbo Station

The Cryo Vac Turbo Station has two buttons that need to be pressed to start the Turbo Station. One is the main power switch inside the hole on the front of the unit and this should be switched on first.

The Display Unit on the front of the Turbo Station then goes through an initialisation process. Once the system has completed its initialisation process, the screen should say:

001: Heating

off

340: XXXX mbar (where XXXX is a pressure reading)

Scroll through to select '**309: Act rotspd**' which will indicate the rotation speed of the turbo when being used. It is recommended that the system is left on this menu item when running.

Press the 'ON' – 'OFF' key on the Turbo Station. This will start the Turbo Station.

Again, like with the Seal Vac Diaphragm Pump, it may be necessary to open the Ballast Valve on the backing diaphragm pump of the Turbo Station, see Figure 11. This will remove any residual water vapour and air in the vacuum lines and body of the PheniX. This is necessary if the vacuum reading begins to rise (or does not drop) during the early stages of pumping down. Open the Ballast Valve for an hour.

The rotation speed of the turbo pump will reach 1500 Hz within 10 minutes. If the turbo does not reach 1500 Hz, it is possible that a vacuum leak exists within the system. The Vacuum System should be stopped and all vacuum connections should be checked to ensure they are clean and sealed properly. If the system fails to reach 1500 Hz, contact Oxford Cryosystems.

Typically, the vacuum pressure should reach 10^{-3} to 10^{-4} mbar within 15 to 20 minutes. The vacuum should be pumped for one hour before the PheniX is switched on and by then, the vacuum should have reached the 10^{-4} to 10^{-5} mbar range.

2.6.4. Turbo station display unit

The display unit on the front of the Turbo Station then goes through an initialisation process. Once the system has completed its initialisation process, the screen should be ready for use. For further information please read the Pfeiffer Vacuum *DCU Display and Operating* Unit Operating Instructions.

1. LCD Display
2. Status Display
3. "Error Acknowledgement" key
4. Key "Left"
5. Key "Right"
6. "Turbo Station ON/OFF" key
7. Red illuminating diode for error status
8. Green illuminating diode for operating status

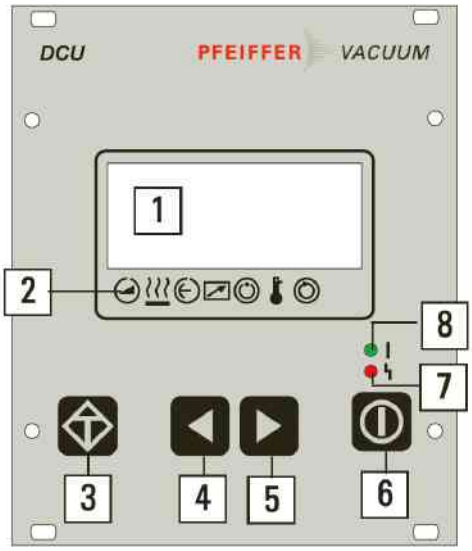


Figure 12 – Turbo station display unit

2.6.5. LED status

The red LED (error status) and green LED (operational status) can take on the following conditions:

Red LED

Illuminates: Collective error messages
Flashes: Warning

Green LED

Illuminates: Mains power unit OK, pumping station ON
Flashes: Mains power unit OK, pumping station OFF
Blinks: Mains power failure

2.6.6. Symbols

By pressing the Left or Right arrow keys, it's possible to scroll through a series of menu options as shown in Table 3

	Symbol	Arrow	Explanation
	Pump acceleration	No arrow	Not accelerating
			Accelerating (flashing)
	Heating	Not in use for PheniX	
	Standby	No arrow	Not in standby mode
			In standby mode
	Under remote control	Not in use for PheniX	
	Switch-point attained (represents	No arrow	Switch-point not attained
			Switch-point attained
	Excessive temperature	No arrow	Temperature OK
			Excessive pump temperature
			Excessive temperature of pump electronics
			Excessive temperature of pump and pump electronics
	Final rotation speed attained	No arrow	Final rotation speed not reached
			Final rotation speed reached

Table 3 – Options on the turbomolecular pumping station

NOTE

The Turbo Station from Pfeiffer is highly configurable and there are many options available to a user of this system. However, the system has been preconfigured by Oxford Cryosystems so there should be no need to adjust the settings of the system.

2.6.7. Switching off the Vacuum System

The system can be stopped by pressing the Turbo Station "ON/OFF" key on the Display Unit.

2.7. Releasing vacuum into the PheniX

IMPORTANT

DO NOT open the vent valve until the Turbo Station has been switched off and has slowed to less than 200 Hz.

To ensure that there is no damage to the Turbo Station or the sample, it is important to release the vacuum in as controlled a way as possible. This can be done by slowly opening the vent valve on the back of the Turbo pump on the Cryo Vac Turbo Station.

Once the PheniX Cooler has warmed up and been shut down it can simply be reprogrammed to start the system up again. IT IS NOT NECESSARY TO SWITCH THE SYSTEM OFF. Simply press START to re-initialise.

IMPORTANT

Open the Vent Valve slowly to prevent damage to the Turbo.

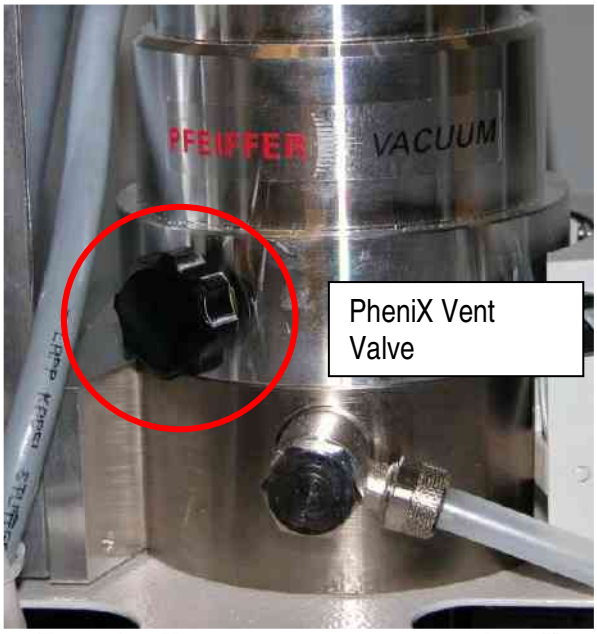


Figure 13 – Venting the vacuum from the PheniX

2.8. Checklist to start the PheniX running

Double check:

- ✓ Cryodrive has adequate water-cooling and switched on.
- ✓ The Turbo Station is connected to the PheniX, switched on and the PheniX has a vacuum of $<10^{-5}$mbar.
- ✓ The controller is switched on.

2.9. Adjusting the height of the PheniX sample stage

The height of the sample stage can be adjusted by turning the red dial on the bottom of the dovetail of the PheniX. There is also a dial on the side to allow the PheniX to be moved horizontally, if necessary.

2.10. How to shut down the PheniX

To shut down the PheniX Cooler correctly, the user should program a WARM phase into the Phase Table. The PheniX will ramp to 310 K, hold that temperature for 30 minutes and then shut down. This procedure ensures that the system is fully warmed up before the vacuum is released.

The PheniX Controller will invite you to switch off the vacuum pump ten minutes before it finishes warming up the Coldhead in order to give the turbomolecular pump time to slow down.

IMPORTANT NOTE

Pressing the red STOP button will immediately turn off the Cryodrive and shut down the heaters within the PheniX Cooler. However, please remember that it may cause damage to the Cooler and Turbomolecular Pump if you then release the vacuum without first ensuring that the system has fully warmed up using the WARM function and the Turbomolecular Pump has stopped completely. This can be checked by looking at the SPEED indicator on the front panel of the pumping outfit

3 PheniX 730 Series Controller

The 730 Series Controller is designed to provide a completely flexible means of controlling temperature. This is achieved by allowing the user to enter up to 8 phases, each one itself providing a complex series of segments. Further phases can be added by using the CryoPad software.

3.1. PheniX controller front panel

The 730 Series PheniX Cooler Front Panel includes the following items

Figure 14 – Front of a Controller

- TEMP screen. Whilst the PheniX Cooler is running this screen displays the temperature of the sample stage. During start-up and shutdown the screen is used for status messages.
- CRYO light: This light is illuminated whenever the Cryodrive compressor is running.
- SPEED meter. The speed meter indicates the current running speed of the Cryodrive compressor.
- STATUS screen. This screen displays information described in detail under the heading *Display Modes*.
- K/°C/°F button and lamps. The button allows the temperature units used by the PheniX to be switched between Kelvin, Centigrade and Fahrenheit **at any time**. The current choice of unit is indicated by the illuminated K/°C/°F lamp. For the purposes of this manual, temperatures are indicated in Kelvin (K).
- SPEED button and lamp. The button allows the Cryodrive speed to be adjusted between a normal value and a faster one. If the SPEED lamp is OFF this indicates normal speed, whereas ON indicates the higher speed. In addition, during a COOL phase the SPEED lamp will flash. This indicates a mode in which the PheniX Cooler fixes the Cryodrive speed at the higher value in order to minimise the cooling time.
- HOLD button and lamp. Pressing the HOLD button will execute a Hold (see *Programming the PheniX*) and illuminate the HOLD lamp. If the PheniX is already in a Hold, pressing HOLD again will release it.
- START button and lamp. The START button switches the PheniX on, executing the start-up phase or the current Phase Table (See *Programming the PheniX, Section 4*). This button is also used to re-start the control program after it has been halted.

- STOP button. The STOP button will immediately halt the PheniX Cooler, turning off the Cryodrive and all the heaters. The Controller may then be switched off, or else re-started by pressing START.

NOTE

It is not safe to release the PheniX Cooler's vacuum or open the cryostat until the system has been warmed up. This can be achieved via a WARM phase, which is the approved method of shutting the system down.

- ALARM button and lamp. If an alarm condition develops (see *Alarm Conditions, Section 4.6*), the ALARM will be illuminated and a buzzer may sound. Pressing the ALARM button will display the cause of the alarm in the STATUS screen, and will also cancel the buzzer.
- DISPLAY button and lamp. The DISPLAY button is used to toggle the Display Mode (see *Display Modes, Section 3.2*), indicated by the corresponding lamp.
- PROGRAM button and lamp. The PROGRAM button is used to toggle Program Mode (see *Programming the PheniX, Section 4*), indicated by the corresponding lamp.
- IntelliKnob. This knob is used to scroll the contents of the STATUS screen. In Program Mode it is also used together with the ENTER button to input information. The IntelliKnob is speed sensitive. This means the faster the Knob is turned, the greater the increment in the number and the slower the Knob is turned, the smaller the increment in the number.
- ENTER button. This button is used during Program Mode (see *Programming the PheniX, Section 4*) to input information.

3.2. Display modes

The STATUS screen displays a variety of information depending on the Display Mode and whether PheniX is **Running** or **Idle**. In each case the contents of the STATUS screen may be scrolled using the IntelliKnob. The various situations are summarised below.

3.2.1. Display Mode 1

PheniX State:
IDLE (Power on, not running)
DISPLAY Lamp: **OFF**
PROGRAM Lamp: **OFF**

Description:
Idle Phase Table Mode
If a program has not been entered, use the IntelliKnob to adjust the temperature and press START to begin. The PheniX will then enter a COOL function and achieve the required temperature as quickly as possible.

If a program has been entered, use the IntelliKnob to scroll the STATUS screen and press START to begin.

Press DISPLAY or PROGRAM to enter the modes below.

3.2.2. Display Mode 2

PheniX State: **IDLE**
DISPLAY Lamp: **ON**
PROGRAM Lamp: **OFF**

Description:
Idle Phase Table

Use the IntelliKnob to scroll through the following information:

Hours	The cumulative time the system has run since manufacture.
Serial#	Controller Serial Number
Software	Version of the Controller software
Shutdown	Indicates the last reason for shutdown. Options are:
STOP	The STOP button has been pressed
END	The system has been shut down due to a programmed END
WARM	The system has been shut down due to a programmed WARM (see *Further Programming on the PheniX Controller, Section 4.2*)
POWER	The power has been switched off at the mains
FLOW	Shutdown due to low flow
TEMP	There has been a large temperature error
SENSOR	This indicates ADC latches or a sensor failure
SINK	Controller Overheating
PSU	Power Supply Overheating
LAST ERR	The last reason for the shutdown but does not include STOP or POWER.

If there has been an unexpected shutdown, the following items are recorded on this list after the LAST ERR:

Set T, Gas T, Cryo Speed, Cryo Status, Shield T, Shield Heat, Cryo Shift, Gas Type, Gas Flow, Pressure, Outer Flow, Nozzle T, Nozzle Heat, Run Time.

These are recorded to allow the user to diagnose the reason for the shutdown and are stored until there is another erroneous shutdown.

Press DISPLAY or PROGRAM to alter the Display Mode.

3.2.3. Display Mode 3

PheniX State: **IDLE**
DISPLAY Lamp: **OFF**
PROGRAM Lamp: **ON**

Description:
Program Mode
This mode allows the user to program the PheniX as described in *Programming the PheniX, Section 4*. The list of phases also gives you the option to save or load a program. 'Save Program' will save the current program and 'Load Program' will load the last saved program.

Press DISPLAY or PROGRAM to alter the Display Mode.

3.2.4. Display Mode 4

PheniX State:
RUNNING
DISPLAY Lamp: **OFF**
PROGRAM Lamp: **OFF**

Description:
Phase Table Mode
The PheniX lists the phases in the current program, with the current phase at the top of the list. The current phase is indicated with a '>'. Use the IntelliKnob to scroll through the list.

Press DISPLAY or PROGRAM to enter the Display Modes 5 & 6.

3.2.5. Display Mode 5

PheniX State:
RUNNING
DISPLAY Lamp: **ON**
PROGRAM Lamp: **OFF**

Description:

Running Display Mode

Set Temp	Temperature
Temp Error	Temperature
Run Time	How long system has been running since START was last pressed.
Gas Type	Detected gas type (He/N2) as displayed via the gas type lamps
Flow Rate	Gas flow in L/min
Pressure	In bar
Outer Flow	Outer gas flow in L/min
Gas Heat	%, the instantaneous value is given and the average is in brackets.
Shield T	Temperature of Cryo Shield brackets
Shield H	%, current Cryo Shield heater power
Nozzle H	%, nozzle heater power (1 or 0). Average value is indicated in brackets.
Nozzle S	Nozzle set point
Nozzle T	Nozzle temperature
Cryo	Cryodrive status
Cryo Speed	Cryodrive speed as a percentage of its maximum.
Cryo Shift	The amount by which the Cryodrive speed has been increased from its normal value in order to achieve rapid cooling.

Press DISPLAY or PROGRAM to alter the Display Mode.

3.2.6. Display Mode 6

PheniX State:
RUNNING
DISPLAY Lamp: **OFF**
PROGRAM Lamp: **ON**

Description:

Program Mode

This mode allows the user to program the PheniX as described in *Programming the PheniX, Section 4*. New phases are added at the end of the list of phases. If the system is in a HOLD, press HOLD to begin the next phase.

Press DISPLAY or PROGRAM to alter the Display Mode.

4 Programming the PheniX

Switch the PheniX Controller on according to the instructions above and wait for the system to initialise. Ensure that the vacuum pump is in the 10^{-4} -10^{-5} mbar range before the PheniX system is used.

4.1. Using the quick start facility and the COOL function

To cool as quickly as possible simply rotate the IntelliKnob to the appropriate temperature and press START. The PheniX will remember the last value requested here and store it for the next time the Quick Start facility is used.

NOTE

The system uses the COOL function to get cold as quickly as possible. The COOL function will automatically set the Cryodrive to its highest speed. This is indicated by the SPEED lamp flashing on and off. Once the system is cold the Cryodrive speed will be gradually reduced towards its normal speed. To maintain the higher speed, press the SPEED button so that lamp remains on permanently.

Once the PheniX reaches the desired temperature, the Controller will automatically enter a HOLD in the Phase Table.

With the system now running, press DISPLAY to view *Display Mode 5* discussed above. This displays all the parameters of the system. Alternatively, press PROGRAM to enter *Display Mode 6* and add additional phases to your program (see *List of Phases* and *Further Programming of the 730 Series Controller* below).

4.2. Further programming of the PheniX Controller

While the system is idle or running, it is possible to program more detailed phases.

Press PROGRAM (this illuminates the PROGRAM lamp). The STATUS screen then displays the following:

Spin the IntelliKnob to see all the phases. Press ENTER at any time to accept a particular phase.

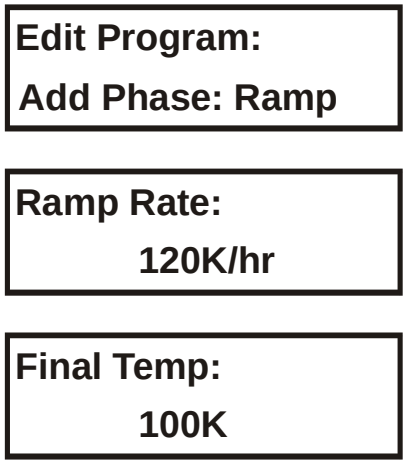
Here is a list of the possible phases and other parameters that each one requires. For an explanation of each phase, see *List of Phases*:

Phase and Description	STATUS Screen Modes

Phase: RAMP

Description
Change temperature at a controlled rate. When ramping down in temperature, if the selected rate is too fast for the PheniX to follow, the Controller will automatically enter the RAMP/WAIT mode (this will be indicated on the screen). The effect of this is to stop the ramp in order for the gas temperature to catch up to within 5 K of the set temperature.

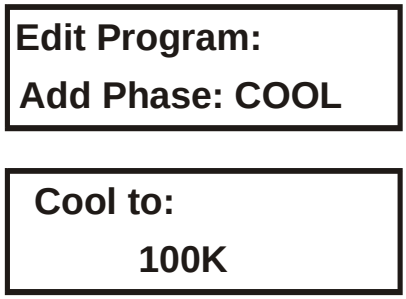
The Ramp Rate may be anything between 1 and 360 K per hour.



Phase: COOL

Description
COOL is designed to get the system as cold as quickly as possible.

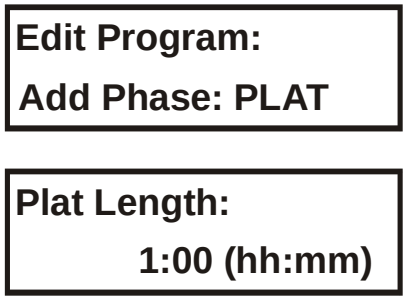
It is not possible to spin the IntelliKnob above the end temperature of the previous phase or the current gas temperature, i.e. you cannot raise the temperature with a COOL.



Phase: PLAT

Description
Maintain temperature fixed for a certain time. The user is prompted to enter a temperature at which to plateau and to specify a time to plateau.

Below 10 hr 00 min the PLAT function will start to count down in seconds and this will be displayed in the STATUS screen during running.

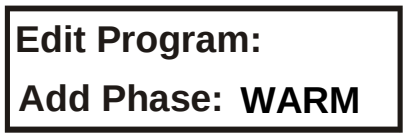


Phase: HOLD

Description
Maintain temperature fixed indefinitely until the START button is pressed (a programmed HOLD should not be confused with the HOLD button).

Phase: WARM

Description
This function is designed to warm up the Coldhead as quickly as possible. It applies maximum power to the heaters in the Coldhead to get to 300 K as quickly as possible. The PURGE is replaced by a SOAKING as the heaters run for a further 10 minutes.



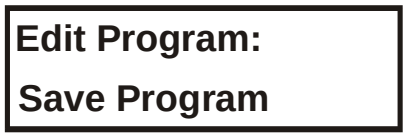
Delete Phase

Description
To delete a phase at any time, in Program Mode, spin the IntelliKnob to 'Delete Phase' and press ENTER. This will delete the last phase entered. If the system happens to be executing this last phase, the phase will be replaced by a HOLD.

Load Program / Save Program

Description
While the system is idle, it is possible to load or save a program. Loading a Program simply loads the last saved program.

Only one program can be stored in the controller memory at a time.



NOTE

Turning the IntelliKnob also offers the option to load a program or save the current program. This is only possible when the system is idle.

Press ENTER on completing each screen. To cancel programming at any time, press PROGRAM or Display, (the PROGRAM lamp will go out).

Once the phases have been entered, press START to begin the first phase in the Phase Table. The Controller will automatically enter a HOLD phase at the end of the program if one has not already been programmed. If the system is running and already in a HOLD phase, pressing the HOLD button will make the program continue to the next instruction in the Phase Table.

4.3. Phase table

Press PROGRAM at any time during running to look at the Phase Table and enter more phases. This will enter *Display Mode 6* but will not give the option of loading or saving a program while the system is running.

If there is a list of phases longer than the screen in the Phase Table, this will be indicated by small characters on the left of the STATUS screen pointing up or down.

↑ ↓	This indicates it is possible to scroll up and down through the phases using the IntelliKnob.
↓	This indicates it is possible to only scroll down through the phases using the IntelliKnob.
↑ ⊥	This indicates that the end of the Phase Table has been reached and it is only possible to scroll up through the phases using the IntelliKnob.
⊤	This indicates that the top of the Phase Table has been reached.
>	This indicates the current programme running.

4.4. Cryodrive speed and the SPEED button

The Cryodrive includes a variable-speed compressor. The speed at which the compressor is running is indicated by the speed meter on the Front Panel. In order to minimise wear, PheniX will automatically reduce the compressor speed to the minimum compatible with maintaining the current set temperature. At all but the very lowest temperatures (<15 K) the compressor will be running at its 'normal' value. Higher speeds may be necessary to achieve temperatures below 15 K.

During a COOL phase PheniX runs the compressor at maximum speed in order to reach the target temperature as quickly as possible. Under these circumstances the lamp above the SPEED button will flash.

Pressing the SPEED button allows the user to over-ride the automatic optimisation of the compressor speed and force the compressor to run at its maximum speed. This is indicated when the lamp above the SPEED button is illuminated steadily. Pressing the SPEED button again cancels this mode.

4.5. HOLD and 'Un-HOLD'

A program can be paused at any time using the HOLD button; this will illuminate the HOLD lamp. To continue the program simply press the HOLD button at any time and the HOLD lamp will go out.

It is also possible to release the HOLD phase by pressing the START button.

4.6. Alarm conditions

The 700 Series style Controller has a number of safety features. If there is an issue with the system, an alarm condition is indicated by an illuminated ALARM lamp and a warning will appear on the bottom screen of the controller. The following list describes all the warnings stored by the Controller.

Temp Warning – If the temperature error (the difference between target temperature and actual temperature) has reach 5K the controller will indicate a warning but will not shut down.

Self-Check Fail – During the initialisation, the controller checks a variety of parameters to make sure that everything is connected properly and that there is continuity in all parts of the system; self-check fail indicates this is not the case. Try restarting the controller a number of times to see if the problem persists. If it does, contact Oxford Cryosystems (support@oxcryo.com).

Temp Control Err – If the temperature error (the difference between target temperature and actual temperature) has reached 25K and the controller reads this value five times from the system, the controller will indicate a warning and shut down.

Temp Reading Err – The controller received a nonsense reading from the temperature sensors.

Sensor Fail – If the controller receives extreme values from the sensors, it will try to reset them. If the sensors fail to reset after five attempts, the controller will shut down with this error.

Brownout – If there is a brief interruption in the electrical supply to the controller, the controller will indicate a 'Brown Out' has occurred. The controller will continue to function normally.

Sink Overheat – If the controller overheats, there will be a Sink Overheat warning. This is often due to the covering of the fan on the underside of the controller.

PSU Overheat – If the controller overheats, there could be a PSU Overheat warning. This is often due to the covering of the fan on the underside of the controller.

Power Loss – When the power to a controller is cut, the controller will report a Power Loss error in the diagnostic screen when it is restarted.

Cryodrive Off? – The controller is connected to the Cryodrive Compressor. If this connection is broken, the Cryodrive is switched off or there is no power to the Cryodrive, the controller will indicate it.

Cryodrive Error – There are a number of warnings given out by the Cryodrive. These include temperature warnings and pressure warnings. A temperature warning may arise due to the water temperature being too hot or too cold. A pressure warning may arise from a low helium pressure.

When there is a warning or if the system shuts down, the ALARM lamp will flash quickly and the buzzer will sound. See the diagnostics by pressing DISPLAY and report these to Oxford Cryosystems.

4.7. Safety features during power failures

The PheniX Controller is designed to protect itself and the sample during power interruptions.

It is possible for the Controller to maintain cooling and not reset the Controller during electrical interruption indicated by the term 'Brown-Out' of between 0-2 seconds. If a 'Brown-Out' is detected, this is indicated on the screen.

4.8. PheniX shut downs

The 730 Series PheniX Controller has been designed to fully protect the PheniX Cooler under its normal mode of operation as outlined in this manual. The Controller will shut down the PheniX Cooler if:

1. The Sample Temp registers an error greater than ±25 K.

2. There is a fault with the Cryodrive. For example, if cooling water is not available, the Cryodrive will overheat and cause PheniX to shut down.

3. The Controller or power supply overheats.

4. A temperature sensor (Sample or Shield) fault occurs.

5. External electrical noise (e.g. from a rotating anode) is too great to allow accurate temperature readings to be taken.

In each case the run time variables are saved (see *Display Mode 3*) and a suitable error message is displayed.

In the case of an unexpected shut down, record the reason for the shutdown as indicated on the STATUS screen and press START to have the option to reprogram it. Pressing START again will reinitialise the Controller. To see a full list of diagnostics at the time of shut down, press DISPLAY which will take you into *Display Mode 2*. Use the IntelliKnob to scroll through the 'Last Shutdown Diagnostics' listed after the 'Last Err' entry.

5 Running the PheniX with Cryopad

Cryopad is a PC program which allows remote monitoring and control of any 700 series Oxford Cryosystems device. This includes the 700 series Cryostream / Plus, Cobra / Plus, PheniX and N-HeliX systems.

5.1. Installing Cryopad

Install Cryopad from the CD also supplied with the Cryostream or download from the Oxford Cryosystems website, www.oxcryo.com/software/cryopad. If you experience problems with the web installer please install the Microsoft Visual C++ Redistributable Package as explained on the above web site.

5.2. Using Cryopad to run the PheniX

With the controller switched off connect a COM port from your PC to the port labelled SERIAL on the back of the controller. **This connection can be made with any standard M-F serial cable**. Now turn on the controller and start Cryopad by selecting from the Oxford Cryosystems group in the Start Menu, or else by double-clicking the Cryopad logo on your desktop.

5.2.1. Connecting using the Settings page

The first time you use Cryopad you will need to select a COM port using the Settings page. If you know which COM port you are using then select it from the **Connect Using Port** menu. If you are using a non-standard COM port you may type its name directly here. Alternatively click the **Search…** button to display the Search dialog, which may be used at any time to scan your computer's COM ports for compatible devices.

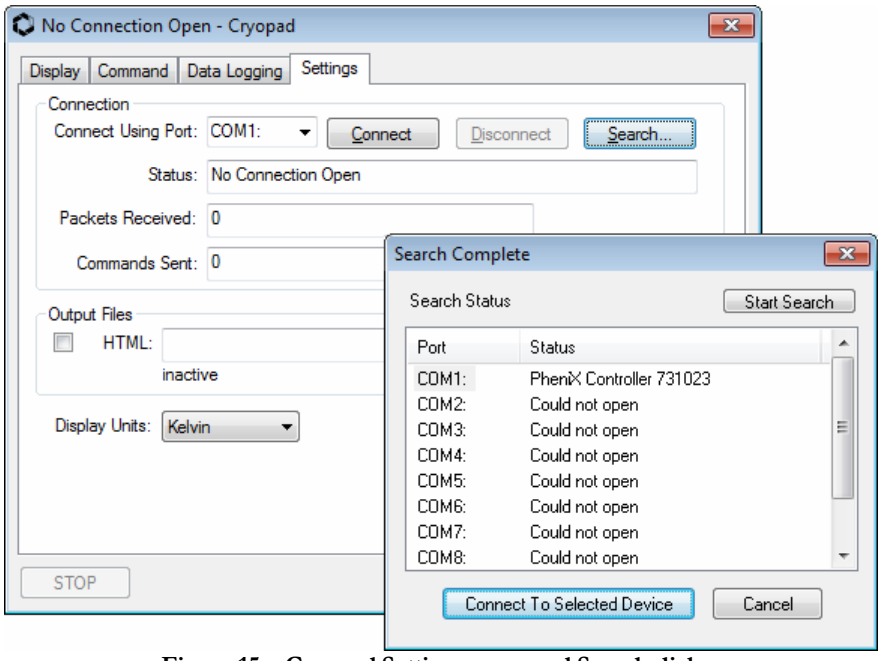


Figure 15 – Cryopad Settings page and Search dialog

The next time you run Cryopad your chosen COM port will be opened automatically, and a connection will be established as soon as a compatible device is detected. If you need to change the COM port, switch to the Settings page, press Disconnect and repeat the above procedure. If you want to connect more than one device, run a new copy of Cryopad for each device and connect each one as described above.

5.2.2. The Display page

Once a connection has been established the Display page shows the live status of the device. The table below indicates the meaning of the various quantities displayed. After half an hour or so when the device has reached its normal operating values all the indicators will appear green.

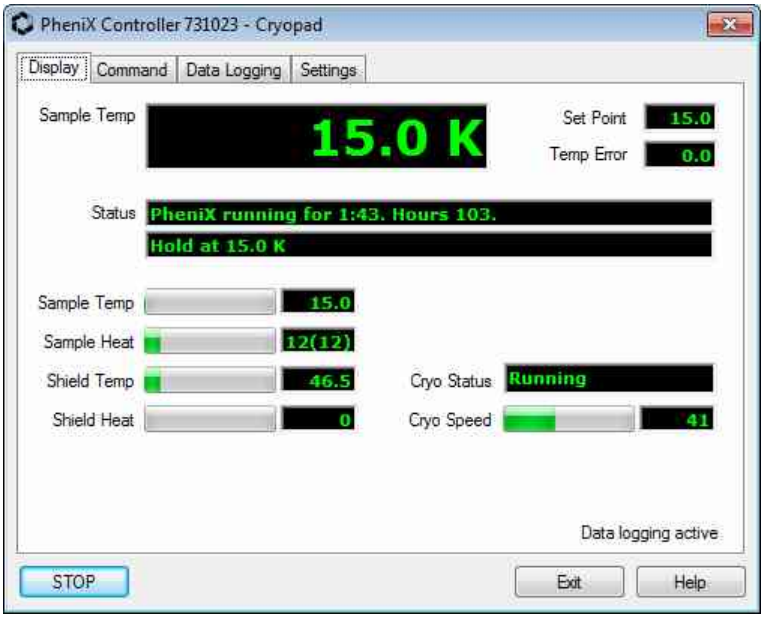


Figure 16 – Cryopad Display page

Data	Explanation
Sample Temp	The temperature at the crystal position
Set Point	The temperature to which PheniX is controlling
Temp Error	The difference between the Sample Temp and the Set Temp, except in a Cool phase, in which Temp Error is zero.
Status	Two lines of information indicating the current status of PheniX. Any errors or warnings raised by PheniX will be displayed here.
Sample Heat	The power to the sample heater, expressed as a percentage of full power.
Shield Temp	The temperature measured by the shield sensor.
Shield Heat	The power to the shield heater, expressed as a percentage of full power.
Cryo Status	An indication of the status of the Cryodrive.
Cryo Speed	The current Cryodrive speed, presented in RPM.

5.2.3. The Command page

The Command page allows commands to be sent to your PheniX exactly as if they were entered using the controller. Commands sent in this way will immediately overwrite the contents of the controller's Phase table. Refer to section 4 above for details of the commands.

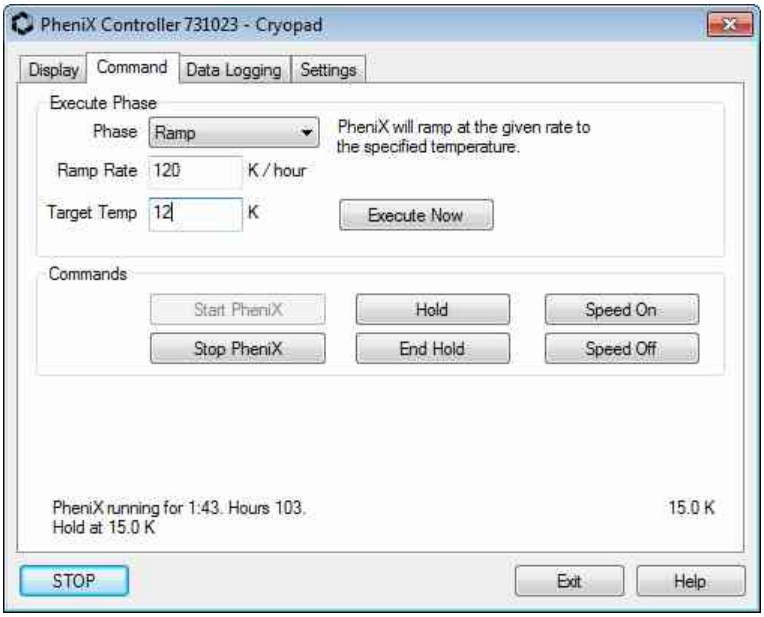


Figure 17 – Cryopad Command page

5.2.4. The Data Logging page

Cryopad allows data to be logged to a tab-delimited text file suitable for use in Excel or similar programs. Choose a file to which the data will be logged using the Log File item, and select the quantities of interest using the check boxes. The Interval item allows you to select the interval in seconds at which the data are logged. For monitoring purposes a 60 s interval is suitable whereas for diagnosing problems an interval of 1 s provides the most information but will produce a larger log file. Check the Logging Active box to commence logging.

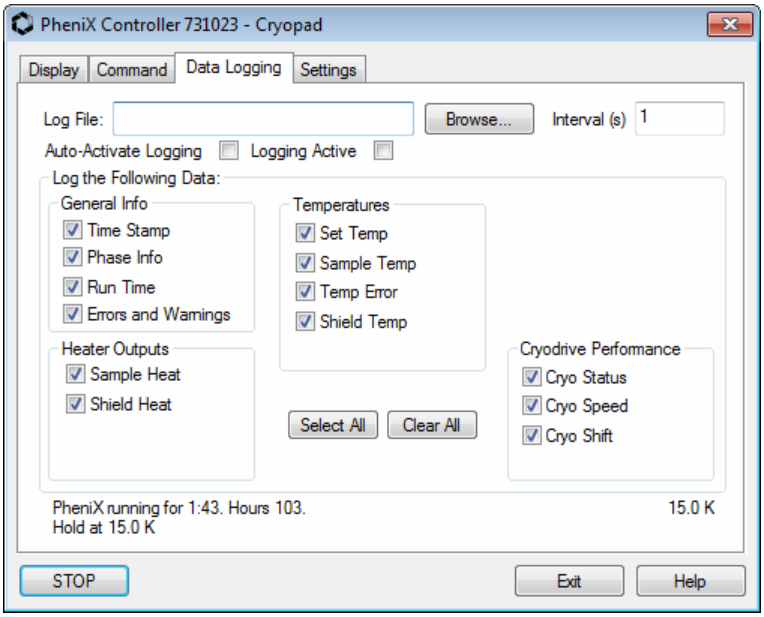


Figure 18 – Cryopad Data Logging page

6 PheniX User Maintenance

The PheniX has been designed to be as easy to use as possible and should run without the need for constant attention, once the user is accustomed to the system. However, some parts of the system will eventually require maintenance, with the following maximum service intervals:

Procedure	Maximum Service Interval
Oil Change For Vacuum Pump[1]	8,000 Hours
Maintenance of Helium level in Cryodrive 3.0[2]	As Required
Change of Diaphragms and Valve In The Diaphragm Pump[3]	10,000 Hours
PheniX Coldhead Swap-Out[4]	15,000 Hours
Replacement of Adsorber in Cryodrive 3.0[5]	15,000 Hours
Change Of Spare Parts Inside Turbomolecular Vacuum Pump[6]	4 Years

Table 4 - PheniX Service Intervals

1. See Section 6.2 for details.
2. See Section 6.4 for details.
3. See Section 6.2 for details.
4. See Section 6.1 for details.
5. See Section 6.3 for details.
6. See Section 6.2 for details.

6.1. Coldhead swap-out

The Coldhead located inside the PheniX will eventually require a service due to the delicate seals and 'O' rings wearing out. If the system begins to have problems reaching base temperature, please contact Oxford Cryosystems for advice and to arrange for a service.

6.2. Servicing the Turbomolecular Vacuum Pumping Station

If the Pfeiffer Turbomolecular Vacuum Pump is supplied, maintenance should be carried out approximately every four year independent of operating hours.

NOTE

If using an alternative pump, the service intervals may be different. Consult your pump manual, or contact the supplier of the pump for information on service intervals and rquired maintenance.

Parts to be serviced:

- Change of operating fluid reservoir
- Change of turbopump bearing

Please contact Oxford Cryosystems for further assistance and purchase of these items. Alternatively you may wish to consult the Operating Manual supplied by Pfeiffer Vacuum Company.

6.3. Replacing the Cryodrive Adsorber

When the Cryodrive has been operating for approximately 16000 hours, you must replace the Adsorber with a new one to avoid permanent damage to the PheniX Coldhead. Please contact Oxford Cryosystems for more information.

The new Adsorber is supplied pressurised with helium, so you should not have to re-charge the Cryodrive with helium after you fit the new Adsorber. A de-pressurisation adapter is supplied with the new Adsorber.

If necessary, refer to the 'Cryodrive Operation & Instruction Guide' for details of how to connect and disconnect the self-sealing Aeroquip coupling used for the Adsorber in the Cryodrive.

WARNING

Do not bend over the internal pipe work when you fit and remove the Adsorber. After removal, the old Adsorber must be safely depressurised before disposal. The replacement Adsorber will be charged with helium to 16.5 bar. Always vent gas safely, directed away from personnel.

Replacing the Adsorber:

1. Switch the Cryodrive ON/OFF switch to OFF. Isolate the Cryodrive from the electrical supply.
2. Remove the lid of the Cryodrive.
3. Disconnect the helium supply hose located at the rear of the Cryodrive.
4. Disconnect the Aeroquip coupling from the Adsorber inlet.
5. Unscrew and remove the Adsorber rear panel locking nut and washer. Remove the single screw retaining the Adsorber inlet connection clamp plate. Lift the Adsorber out of its locating hole and remove the Adsorber. Remove the locking nut and clamp plate from the Adsorber inlet connection. Retain the clamp plate and screw, locking nut, washer and star washer.
6. Depressurise the old Adsorber by connecting the depressurisation adaptor to the Adsorber helium inlet and outlet coupling and tightening slowly by hand.
7. Remove the dust from the inlet and discharge self-sealing couplings of the new Adsorber. Fit the Adsorber clamp plate and locking nut to the Adsorber Aeroquip inlet connection.
8. Install the new Adsorber in position in the compressor unit and ensure that the locating pin is correctly engaged. Secure the new Adsorber in place using the nut and washers, and Adsorber clamp plate screw retained in step 5.
9. Re-connect the helium supply hose. Re-connect the internal Aeroquip on the Adsorber inlet.
10. Re-fit the lid of the Cryodrive.

Check that the pressure gauge reads 16.5±1.0 bar (239.31±14.5 psig). If the gauge reads below 15.5 bar, add helium gas following the procedure detailed in Section 6.4.

►Record the date the new Adsorber is fitted. Also record the hours run from the PheniX controller hour counter.

6.4. Topping up the Cryodrive with helium

The PheniX system involves a closed helium gas circuit. The helium in this circuit will eventually leak out over time and will require topping up from a high pressure helium gas cylinder (99.9995%). **Note: that this should only be performed when the system is off.**

Re-charge the Cryodrive with helium if the helium pressure falls to below 15.5 bar.

CAUTION

Ensure that the interconnecting pipe work is capable of safely withstanding the maximum regulator delivery pressure.

WARNING

When you vent helium from the charge and vent adapter during the purging procedure, ensure that the vented gas is directed safely away from personnel.

CAUTION

You must re-charge the Cryodrive with 99.9995% helium. If you do not, you will contaminate the Cryodrive with impurities that will reduce its efficiency.

Re-charge the Cryodrive with helium if the helium pressure falls to below 15.5 bar. If you need to re-charge the Cryodrive frequently (for example, every 6 months or more often), there is probably a leak in your installation. Use a helium leak detector or other suitable method to find the leaks, and contact Oxford Cryosystems with the results, or for further guidance.

To recharge the system with Helium, please follow the instructions in Section 5.3 of the Cryodrive manual, supplied with the Cryodrive.

Note that if the helium pressure has fallen to 0 bar (atmospheric pressure), the helium should not be topped up. Instead, contact Oxford Cryosystems for advice on how to proceed.

7 Liquid and gaseous nitrogen safety sheet

7.1. General

These safety points are a guideline to outline the potential hazards and procedures involved in the handling of liquid or gaseous nitrogen. Anyone handling liquid or gaseous nitrogen should first inform their departmental or laboratory safety advisor and receive advice about local safety procedures.

All users are requested to read this safety sheet before handling the PheniX. Oxford Cryosystems accept no responsibility for injury or damage caused by the mishandling of liquid or gaseous nitrogen.

7.1.1. General properties

- Gaseous nitrogen is colourless, odourless and tasteless and is slightly lighter than air at equal temperatures; cold nitrogen vapour is, however, denser than atmospheric air.
- Liquid nitrogen is odourless, colourless and boils at -195.8°C. One volume of liquid nitrogen gives approximately 700 volumes of gas at ambient conditions.
- Nitrogen is not flammable. It is chemically inert, except at high temperatures and pressures. Its volume concentration in air is 78%.
- Liquid and cold gaseous nitrogen can cause severe burns or frostbite when in contact with the skin or respiratory tract.
- Gaseous and liquid nitrogen is non-corrosive.
- Nitrogen does not support life and acts as an asphyxiant.
- Nitrogen is intrinsically non-toxic.

7.2. Fire and explosion hazards

Gaseous and liquid nitrogen are non-flammable and do not, themselves, constitute a fire or explosion risk. However, both gaseous and liquid nitrogen are normally stored under pressure and the storage vessels whether gas cylinders or liquid tanks, should not be located in areas where there is a high risk of fire or where they may normally be exposed to excessive heat.

7.3. Health hazards

7.3.1. Asphyxia

Nitrogen, although non-toxic, can constitute an asphyxiation hazard through the displacement of the oxygen in the atmosphere. Nitrogen gas or oxygen depletion is detectable by the normal human senses.

Oxygen is necessary to support life and its volume concentration is the atmosphere is 21%. At normal atmospheric pressure persons may be exposed to oxygen concentrations of 18% or even less, without adverse effects. However, the response of individuals to oxygen deprivation varies appreciably. The minimum oxygen content of breathing atmospheres should be 18% by volume but to ensure a wider margin of operational safety it is recommended that persons are not exposed to atmospheres in which the oxygen concentration is, or may become, less than 20% by volume.

Symptoms of oxygen deprivation, such as increased pulse and rate of breathing, fatigue, and abnormal perceptions or responses, may be apparent at an oxygen concentration of 16%.

Permanent brain damage or death may arise from breathing atmospheres containing less than 10% oxygen. Initial symptoms will include nausea, vomiting and gasping respiration. Persons exposed to such atmospheres may be unable to help themselves or warn others of their predicament. The symptoms are an inadequate warning of the hazard.

WARNING

Breathing a pure nitrogen atmosphere will produce immediate loss of consciousness and almost immediate death.

7.3.2. Cold burns

Liquid and cold nitrogen vapours or gases can produce effects on the skin similar to a burn. Naked parts of the body coming into contact with un-insulated parts of equipment may also stick fast (as all available moisture is frozen) and the flesh may be torn on removal.

7.3.3. Frostbite

Severe or prolonged exposure to cold nitrogen vapour or gases can cause frostbite. Local pain usually gives warning of freezing but sometimes no pain is experienced. Frozen tissues are painless and appear waxy with a pallid yellowish colour. Thawing of the frozen tissues can cause intense pain. Shock may also occur if the burns are at all extensive.

7.3.4. Effect of cold on Lungs

Prolonged breathing of extremely cold atmospheres may damage the lungs.

7.3.5. Hypothermia

Low environmental temperatures can cause hypothermia and all persons at risk should wear warm clothing. Hypothermia is possible in any environmental temperature below 10ºC but susceptibility depends on time, temperature and the individual. Older persons are more likely to be affected. Individuals suffering from hypothermia may find that their physical and mental reactions are adversely affected.

7.4. Precautions

7.4.1. Operations and maintenance

It is essential that operations involving the use of gaseous or liquid nitrogen, particularly where large quantities are used, are conducted in well-ventilated areas to prevent the formation of oxygen deficient atmospheres.

Ideally, nitrogen should be vented into the open air well away from areas frequented by personnel. It should never be released or vented into enclosed areas or buildings where the ventilation is inadequate. Cold nitrogen vapours are denser than air and can accumulate in low lying areas such as pits and trenches.

Where large spills of liquid nitrogen occur a fog forms in the vicinity of the spill caused by the condensation of water vapour in the surrounding air. The fog, in addition to severely reducing visibility, may contain oxygen concentrations appreciably lower than that of the air presenting a local asphyxiation hazard.

7.4.2. Personnel protection

Persons handling equipment in service with liquid nitrogen should wear protective face shields, loose fitting gauntlets and safety footwear.

7.4.3. Emergencies

In the event of an accident or emergency the instructions below should be implemented without delay.

7.4.4. Asphyxiation

Persons showing symptoms of oxygen deprivation should be moved immediately to a normal atmosphere. Persons who are unconscious or not breathing must receive immediate first aid. Medical assistance should be summoned without delay. First aid measures included inspection of the victim's airway for obstruction, artificial respiration and simultaneous administration of oxygen. **These procedures should only be carried out by a trained first aid staff.** The injured should be kept warm and resting.

It is important that the personnel carrying out rescue operations should minimise the risk to themselves.

7.4.5. Treatment of cold burns and frostbite

Cold burns should receive medical attention as quickly as possible. However, such injuries are not an everyday occurrence and doctors, hospital staff or works first aid personnel may not be aware of the basic methods of treatment. The following notes describe the first aid treatment and recommended advice for further treatment to be given by a medical practitioner or a hospital.

7.5. First aid

In severe cases summon medical attention immediately. Flush affected areas of skin with copious quantities of tepid water to reduce freezing of tissue. Loosen any clothing that may restrict blood circulation. Move the victim to a warm place but not to a hot environment and do not apply direct heat to the affected parts. Every effort should be made to protect frozen parts from infection and further injury. Dry, sterilised bulky dressings may be used but should not be applied so tightly that blood circulation is restricted.

7.5.1. Treatment by medical practitioner or hospital

1. Remove any clothing that may constrict the circulation to the frozen area. Remove patient to sick bay or hospital.

2. Immediately place the part of the body exposed to the cryogenic material in a water bath which has a temperature of not less than 40°C but no more than 45°C. **Never use dry heat or hot water.** Temperatures in excess of 45°C will superimpose a burn upon the frozen tissue.

3. If there has been a massive exposure to the super cooled material so that the general body temperature is depressed, the patient must be re-warmed gradually. Shock may occur during re-warming, especially if this is rapid.

4. Frozen tissues are painless and appear waxy with a pallid yellowish colour. They become painful, swollen and very prone to infection when thawed. Therefore, do not re-warm rapidly if the accident occurs in the field and the patient cannot be transported to hospital immediately. Thawing may take from 15-60 minutes and should be continued until the blue, pale colour of the skin turns to pink or red. Morphine, or some potent analgesic, is required to control the pain during thawing and should be administered under professional medical supervision.

5. If the frozen part of the body has thawed by the time medical attention has been obtained, do not re-warm. Under these circumstances cover the area with dry sterile dressings and a large bulky protective covering.

6. Administer a tetanus booster after hospitalisation.

7.5.2. Hypothermia

Persons suspected to be suffering from hypothermia should be wrapped in blankets and moved to a warm place. Slow restoration of temperature is necessary and forms of locally applied heat should not be used. Summon medical attention.

7.5.3. Liquid nitrogen spillage

If large spills of liquid nitrogen occur, large quantities of water should be used to increase the rate of liquid vaporisation.

8 Technical Support

To allow Oxford Cryosystems to offer fast and accurate technical support, please quote your PheniX Serial Number with all technical issues. It is worth keeping a record of this number in a convenient place:

PheniX SERIAL NUMBER
This PheniX serial number is _____

Before you return your equipment you must warn Oxford Cryosystems by contacting us.

Oxford Cryosystems Ltd contact details:

Email: support@oxcryo.com

Phone: +44 (0)1993 883488

Fax: +44 (0)1993 883988

8.1. Returns procedure

Use the following procedure to return ANY items for repair.

1. Contact Oxford Cryosystems and obtain an 'RMA' number for your equipment which must be written on each box that you return. Without this number we may reject packages. You will also be emailed a form which you must fill in and email or fax back to us prior to sending your package(s).

2. Remove all traces of dangerous substances and any accessories that will be returned to Oxford Cryosystems. Drain all fluids and lubricants from the equipment and its accessories.

3. Disconnect all accessories from the equipment. Safely dispose of the filter elements from any oil mist filters.

4. Seal up all of the equipment's inlets and outlets (including those where accessories were attached). You may seal the inlets and outlets with blanking flanges or heavy gauge PVC tape.

5. Seal contaminated equipment in a thick polythene bag. If you do not have a polythene bag large enough to contain the equipment, you can use a thick polythene sheet.

6. If the equipment is large, strap the equipment and its accessories to a wooden pallet. Preferably, the pallet should be no larger than 510 mm x 915 mm (20"x 35"); contact Oxford Cryosystems if you cannot meet this requirement.

7. If the equipment is too small to be strapped to a pallet, pack it in a suitable strong box.

8. If the equipment is contaminated, label the pallet (or box) in accordance with laws covering the transport of dangerous substances.

Oxford Cryosystems - Warranty Certificate

This warranty is subject to the Oxford Cryosystems Ltd (OCL) Terms and Conditions of Sale.

OCL warrants to the Buyer that the goods sold for use hereunder will be free from defects in material and workmanship under normal use and operation for 12 months from the date of shipment from OCL's premises.

In order to obtain the benefits of the warranty the Buyer must first notify OCL of the defects. An OCL representative will verify the nature of the defect and if it is covered by this warranty, OCL will issue the Buyer with a RMA number and provide the Buyer with instructions on how to return the goods to OCL. The Buyer must return the goods according to instructions from OCL, complete with a written description of the claimed defect and RMA number. The goods should be packed safely, preferably in its original packaging prior to return.

The Buyer shall meet the cost of shipping the defective goods to OCL and OCL will pay any return costs to the Buyer

OCL's obligation under this warranty is limited to its option to repair or replace goods that are proven to be defective when used under normal operating conditions and within specification.. This warranty does not cover any changes made by the customer, depreciation of the goods or claims for compensation.

No warranty is given for damage resulting from misuse or fair wear and tear. In addition, this warranty does not cover any costs incurred in damage arising from the dismantling or reassembly of any of the goods, or for consequential losses of time or materials caused by Cryostream failure.

Registration
In order for us to be able to provide fast and effective service, you should register your system with us. Please send the serial number of the system (found engraved on the Coldhead) to support@oxcryo.com, together with your full contact details.

To make contact with Oxford Cryosystems you can telephone, fax, or email us at:

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Email: Info@Oxcryo.com